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DISPERSION-STRENGTHENED NICKEL-ALUMINA ALLOY PRODUCED FROM COMMINUTED POWDERS

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16. Abstract

An investigation was conducted to determine whether a nickel - 2-volume-percent alumina dispersion-strengthened material with a fine, uniformly distributed dispersoid could be produced which was equivalent in short-time tensile strength to commercially available thoriated sheet materials. Comminution and blending with a modified triple-stirrer attritor and a hydrogen and vacuum precleaning treatment prior to consolidation were used. A product with a fine dispersoid with an average particle size of 0.04 μm and an interparticle spacing of 0.7 μm was achieved. This material had a 1093° C (2000° F) short-time tensile strength of 117 MN/m^2 (16 900 psi).

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DISPERSION-STRENGTHENED NICKEL-ALUMINA ALLOY PRODUCED FROM COMMINUTED POWDERS

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SUMMARY

This investigation was conducted to determine if by improved processing a nickel-alumina (NI-Al_2O_3) dispersion-strengthened material with a good microstructure and short-time tensile strength comparable to commercially available thoriated sheet materials could be produced.

Nickel - 2-volume-percent-alumina composites were prepared by mechanical comminution and blending of the powders. The products were precleaned, cold-pressed, sintered, hot-rolled, and subjected to 7, 14, or 21 cold-roll - anneal cycles to develop the strength of the material. A nickel - 2-volume-percent-alumina material produced in a previous NASA study was used as a reference. A Ni-Al_2O_3 sheet material produced for this study had a better dispersoid distribution and was superior to the reference material and compared favorably with commercially available thoriated sheet products in short-time 1093° C (2000° F) tensile strength.

The best product made had an average alumina particle size of 0.04 micrometer, interparticle spacing of 0.7 micrometer, and a tensile strength at 1093° C of 117 meganewtons per square meter (16 900 psi). The achievement of a material with good properties was due in part to the use of a triple-stirrer attritor (with a bottom tap) for grinding the powders and the use of a modified hydrogen and vacuum precleaning treatment to reduce the presence of detrimental impurities.

INTRODUCTION

One method of enhancing the high-temperature strength of materials is by the addition of fine, stable oxide dispersoids. Alumina (Al_2O_3) as a dispersoid has been successfully used in materials such as aluminum and copper alloys. Higher temperature materials such as nickel and nickel-chromium alloys have been made most successfully with thoria (ThO_2) additions. The first commercially produced high-temperature nickel (refs. 1 to 3) contained 2 percent ThO_2. This material is now in use in various high-

temperature applications. The thoria has a greater thermodynamic stability than does alunima and is more stable in metal matrices. Numerous examples have been given in the literature which show thoria in nickel to be extremely stable at temperatures as high as 1315° C (2400° F) (ref. 4). In reference 4 evidence was also obtained to show that alumina had a high degree of stability at high temperatures, although it did agglomerate to some extent at the very high temperatures of 1260° to 1425° C (2300° to 2600° F).

Although good properties have been achieved with thoria-dispersion-strengthened nickel (TD-Ni), some advantages might accrue from the use of alumina rather than thoria as a dispersoid. One of these has to do with the fact that materials made with thoria are slightly radioactive, while those made with alumina are not. Another is that alumina is less expensive than thoria and requires no special precautions in processing. For these reasons it was felt that attempts to produce alumina dispersion-strengthened materials by processes utilized at NASA with thoriated nickel should be undertaken.

Preliminary experiments at the Lewis Research Center have shown that it is possible to produce better dispersions by comminution and blending with a modified triple-stirrer attritor than with a single-stirrer attritor. In addition, evidence has been obtained that, if the procedure for cleaning and removing matrix oxides from compacts is varied, enhanced strengthening of dispersion materials may be possible.

The object of this investigation, then, was to determine whether improved processing procedures would produce a nickel - 2-volume-percent-alumina dispersion-strengthened material with finer, more uniformly distributed dispersoid particles and smaller interparticle spacing and whether such a material would have a 1093° C (2000° F) tensile strength comparable to that of commercial thoriated material.

Nickel with 2 volume percent alumina was produced by grinding in a triple-stirrer attritor and processed by methods previously developed at NASA. This consisted essentially of precleaning, consolidating, sintering, and thermomechanical processing by rolling with intermediate anneals. The material was then subjected to short-time tensile tests at 1093° C. A nickel - 2-volume-percent-alumina material produced in a previous study (ref. 5) was used as a standard of comparison with that produced in the current investigation.

MATERIALS, APPARATUS, AND PROCEDURE

Materials

The raw material used in this investigation consisted of 2.5-micrometer 99.7-percent-pure Inco-B carbonyl nickel powder and 0.03-micrometer Alon C alumina powder.

Apparatus

The grinding apparatus used was a triple-stirrer attritor. Other apparatus used included a conventional cold press, a cold isostatic press, a furnace for cleaning and sintering the compacted powder, and a vacuum tensile testing machine capable of testing materials at a vacuum of 10^{-5} torr. Hot-rolling was done on a four-high mill with 6.4-centimeter-diameter by 20-centimeter-long working rolls at a surface speed of 82.4 meters per minute. Cold-rolling was done on a four-high mill with the same size rolls as the hot rolls at a surface speed of 1.4 meters per minute. Inductively heated high-purity hydrogen furnaces were used for the hot-rolling and intermediate annealing operations.

Procedure

The processing steps may be summarized as follows:

(1) Preparation of fine powder blend - grinding of nickel and alumina in heptane and alcohol for 69 hours

(2) Densification

- (a) Powder is heated to 315° C (600° F) in hydrogen (with or without vacuum treatment).
- (b) Powder is cold-pressed into slabs (2.5 by 7.5 by 0.4 cm; 1.0 by 3.0 by 0.15 in.) at 55 meganewtons per square meter (8000 psi) in argon.
- (c) Slabs are cold-pressed at 480 meganewtons per square meter (70 000 psi) in isostatic press.
- (d) Slabs are heated to 1093° C (2000° F) in hydrogen and held for minimum of 4 hours.

(3) Thermomechanical processing

- (a) Slabs are hot-rolled two passes at 30 percent reduction per pass at 1093° C (2000° F).
- (b) Slabs are cold-rolled 7, 14, or 21 passes at 10 percent reduction per pass (with intermediate anneals of 30 min at 1204° C (2200° F) in hydrogen).

The specimens were stamped from the rolled sheets and tensile tested after 7, 14, and 21 cold-roll - anneal cycles. The methods used with a few modifications are similar to those described in reference 5.

Grinding. - The nickel and alumina powders were ground in an attritor in a heptane - 10 percent ethyl alcohol medium. A triple-stirrer attritor was used for the grinding. This machine grinds more efficiently than the single-stirrer attritor used to grind the $Ni\text{-}Al_2O_3$ material prepared in the previous study (ref. 5). In addition, the triple-

stirrer attritor has a bottom tap valve which facilitates separation of the slurry from the balls. This reduces the amount of time and handling required in unloading an attritor, which is ordinarily done by dumping the slurry and balls from the top onto a screen. Nickel balls, nickel stirrers, and a nickel container were utilized to avoid contamination of the powders. During grinding an argon blanket was used over the top of the slurry to minimize oxidation of the powder. The slurry was ground for 69 hours, after which time the average particle size was about 300×10^{-10} meter (300 Å).

When grinding was completed, the slurry was permitted to drain out of the bottom tap into a stainless-steel container. The powder in the slurry was allowed to settle 15 minutes, and then the bulk of the material was siphoned into another container. The large particles which had settled to the bottom were left behind in the original container and were discarded. The powder in the siphoned slurry was again allowed to settle. Now the clear supernatant liquid was decanted, and the remaining heptane and alcohol was permitted to evaporate in a hood so that an easily breakable powder cake was left for further processing.

Precleaning or annealing of powders. - The dried powder cake was divided into three portions and precleaned as shown in table I.

Densification and sintering of slabs. - After precleaning, 20 to 30 grams of powder were cold-pressed into a slab 2.5 by 7.5 by 0.4 centimeter (1.0 by 3.0 by 0.15 in.) in an argon-filled dry box at a nominal load of 56 meganewtons per square meter (8000 psi). The slabs were then sealed in a flexible plastic bag and cold-pressed isostatically at 490 meganewtons per square meter (70 000 psi). The pressed pieces were cleaned and sintered by raising their temperature slowly in hydrogen in a tube furnace to 1093° C (2000° F). The heating rate was controlled so that the monitored effluent moisture level was maintained below 100 ppm. The heating rate was nominally 100° C per hour. Between 4 and 8 hours were required to bring the moisture level to a minimum value. After sintering, the density of the slabs was approximately 80 to 85 percent of theoretical.

Thermomechanical working. - The partially densified slabs were heated in hydrogen and were given two hot rolls at 1093° C (2000° F) with reductions of 30 percent each so that the total hot reduction was approximately 50 percent. This was followed by 7, 14, or 21 cold-reduction passes of about 10 percent each with intermediate anneals (after each pass) of 30 minutes in hydrogen at 1204° C (2200° F). After being worked the specimens ranged in thickness from 0.013 to 0.10 centimeter (0.005 to 0.040 in.), depending on the number of passes they had received.

Testing. - Specimens were stamped from the as-rolled sheet and tensile tested at 1093° C (2000° F). Previous work with as-stamped specimens has indicated no significant difference in properties from those which were ground. The specimen configuration used in the tensile test is shown in figure 1. Tensile tests were run at a crosshead rate of 0.05 centimeter per minute (0.02 in./min) in a vacuum of approximately 10^{-5} torr.

The ultimate tensile strength was used to screen the effects of the various processing techniques. Although the determination of other mechanical properties such as stress-rupture and creep is desirable, it was beyond the scope of this program.

Metallographic examination. - Electron micrographic examination of a number of as-rolled specimens was made by using conventional replication and thin-foil transmission. In addition, selected specimens were examined by using a metallographic light microscope.

Pole figures. - A pole-figure diffractometer was used to determine the (111) pole figure of selected specimens from this and a previous investigation. Nickel-filtered copper radiation was used for the Schultz method (ref. 6). The intensity distribution was scanned with a proportional counter along a helical path over a 360° azimuthal range and 0° to 80° inclination. No corrections, other than for background, were made.

RESULTS

Tensile Test Results at 1093° C

Results of the 1093° C (2000° F) tensile tests are given in table II. Tensile strength is plotted against the number of cold-roll - anneal cycles in figure 2. Included in this figure are the data from the previous study (ref. 5), which are used as a basis for comparison. The 1093° C (2000° F) tensile strength of the triple-stirrer-attritor-ground, hydrogen-cleaned Ni-Al_2O_3 of this study (material B) was considerably greater than that of the single-stirrer-attritor-ground, hydrogen-cleaned Ni-Al_2O_3 of the previous study (material A) after 7 and 14 cold-roll - anneal cycles. The increased tensile strength was approximately 35 meganewtons per square meter (5000 psi) after 7 cycles and 11.2 meganewtons per square meter (1600 psi) after 14 cycles. Extrapolating the data for material A from 18 to 21 cycles shows the tensile strength at this point to be approximately the same as for materials B and C. For the few data points obtained, less scatter was observed for material B than material A.

The Ni-Al_2O_3 precleaned in hydrogen followed by vacuum (material C) proved to have the greatest strength obtained in the investigation with a 1093° C (2000° F) tensile strength of 117 meganewtons per square meter (16 900 psi). An interesting feature of the tensile curve for the hydrogen-vacuum precleaned material (C) as compared with the curve for the hydrogen-cleaned material (B) is that it was lower after 7 cycles but higher after 14 cycles. Thus, the slopes of the two curves are quite different. In both cases, however, the maximum 1093° C (2000° F) tensile strength was achieved after 14 cold-roll - anneal cycles.

One additional experiment in which the precleaning treatment of the Ni-Al_2O_3 ma-

terial consisted of a 1-hour vacuum treatment at 315° C (600° F) followed by a 1-hour hydrogen treatment at the same temperature (material D) did not show any improvement in 1093° C (2000° F) tensile strength over that for the material receiving the standard 2-hour hydrogen treatment.

Microstructures

Figure 3 shows the replicated microstructure of rolled specimens of Ni-Al_2O_3 ground in a triple-stirrer attritor and precleaned in hydrogen and vacuum compared with the microstructure for Ni-Al_2O_3 ground with a single-stirrer attritor and precleaned in hydrogen. It can be readily seen from the microstructure that the former material (shown in fig. 3(a)) has a much finer particle size and a more uniform particle distribution. The Al_2O_3 particles shown in figure 3(a) have an average particle size of 0.04 micrometer, and the interparticle spacing is approximately 0.7 micrometer, whereas the particles shown in figure 3(b) have an average size of 0.09 micrometer and an interparticle spacing of 3.0 micrometer. This may very well account in part for the better 1093° C (2000° F) tensile properties observed for the triple-stirrer-attritor-ground product.

Further evidence of the good distribution obtained with the triple-stirrer-attritor-ground Ni-Al_2O_3 material with the hydrogen and vacuum precleaning can be seen from the thin-film electron transmission micrographs in figure 4. Figure 4 shows the microstructure after 7, 14, and 21 cold-roll - anneal cycles. Figure 4(b) is a transmission micrograph of 14-cycle material, which is the same material shown in figure 3(a), and this again shows the fine particles and uniform distribution of the Al_2O_3 dispersoid in this product. The transmission micrographs also reveal that in this material a better distribution and larger grain size is present in the stronger 7- and 14-cycle material than in the weaker 21-cycle material. Thus, large grains as well as good distribution appear to improve the tensile properties of the worked material.

Examination of this material by using the light microscope and conventional metallographic techniques did not reveal the grain size or any significant aspects of the microstructure.

Pole figures were determined for material C after 7, 14, and 21 cold-roll - anneal cycles and material A after 7, 14, and 18 cold-roll - anneal cycles. All have essentially the same texture, (011) planes in the [100] direction. On the basis of these results it would appear that differences in tensile strength are not attributable to differences in texture.

DISCUSSION

The results of this investigation have shown that dispersion-strengthened Ni-Al_2O_3 products can be produced from mechanically comminuted and blended powders with tensile properties at 1093° C (2000° F) equivalent to those of commercially produced Ni-ThO_2 sheet products (ref. 7). Generally, the products in this study containing 2 percent Al_2O_3 were superior in tensile properties to those produced by utilizing 2 percent Al_2O_3 in a previous investigation.

Several items of interest in the study which helped to achieve the good properties, particulary those involving processing variables and thermomechanical working, are worthy of note and further discussion.

Significance of Processing Variables

The achievement of a product with a fine microstructure was due in part to the use of novel processing techniques. One of the processing variables involved the use of vacuum following the initial hydrogen precleaning of the powders. The best properties were obtained by using this precleaning treatment (fig. 2, material C). Very probably, the successful results obtained by using vacuum after the hydrogen precleaning were due to the removal of impurities (gases) from the interstices of the powder, particularly water vapor formed by the reaction of the hydrogen with the nickel oxide. Evidence supporting this mechanism is given by the tensile results shown in figure 2 (only two data points) for material D, for which the precleaning treatment used the vacuum prior to the hydrogen. The fact that the 1093° C tensile results for material D in figure 2 are lower than for material C is indicative of the fact that a significant improvement is made by utilizing the vacuum treatment following the hydrogen treatment rather than vice versa and rather than not using a vacuum treatment at all (fig. 2, material B).

Further improvement in material properties may have resulted from the incorporation of a tap valve in the bottom of the triple-stirrer-attritor slurry container. With this valve the ground slurry can be drained and separated from the grinding balls rapidly. This process can be done in 4 to 8 hours compared to the 4 to 5 days required by the previous practice with the single-stirrer attritor without the bottom tap. With a shorter handling time less segragation of the fine powder blend and agglomeration of the oxide dispersoid would be expected.

Another processing variable involved siphoning off the fine powder suspended in the slurry and discarding the coarse powder which settled to the bottom of the container. This separation of the fine particles from the coarse particles was probably also helpful in obtaining an improved product. While it is difficult to determine exactly the extent of

the improvement achieved with each of the processing steps, it was certainly established that the microstructure of the high-strength Ni-Al_2O_3 material produced in this study was superior to that of the preceding study, both in particle size and in interparticle spacing.

Effect of Thermomechanical Working on Tensile Properties

It has already been noted that in this investigation the 1093° C tensile properties for the Ni-2Al_2O_3 materials reached their peak value at 14 cold-roll - anneal cycles. This was also true of the Ni-2Al_2O_3 produced in the previous investigation (ref. 5). However, with the Ni-4ThO_2 products produced in the previous investigation, the tensile properties did not peak at 14 cycles but increased steadily up to 21 cycles. Probably, the quantity of oxide added affects the response to the thermomechanical processing treatments used. The fact that the tensile strength of the Ni-Al_2O_3 composities of both the previous and present investigations peaked at 14 cycles indicates that for these products this is the optimum amount of working. The reason for the successful strengthening as a result of variations in the number of work-anneal cycles is not fully understood. In general, strengtheneing is interrelated with the nature, concentration, and distribution of the oxide and the nature of the fabrication process. While this investigation was not intended to show the relation of thermomechanical processing to volume percent oxide, the work established that Ni-Al_2O_3 composites can be produced with tensile properties comparable to those of commercial thoriated sheet products. Additional experiments on the effect of cold-roll - anneal cycles on the tensile properties of Ni-4Al_2O_3 and Ni-2ThO_2 could help to determine the relative significance of the nature and concentration of the oxide to the response of a dispersion material to thermomechanical processing.

Relation Between Microstructural Parameters and Strength

The particle size and interparticle spacing of the alumina clearly affected the tensile properties of the product. The material produced in this investigation which had the better distribution of finer oxides had higher tensile strength than the material produced in the earlier investigation. The average alumina particle size for the specimens of this study was 0.04 micrometer, and the interparticle spacing was approximately 0.7 micrometer. A slight nonuniformity in distribution was present since larger particles were observed at the surfaces of the specimen. However, the overall dispersoid parameters may be even better than indicated, since many of the smallest particles,

known to be present from the transmission micrograph, are not resolved at the low magnification (X12 000) used in the replicas. In the material produced in the previous investigation the alumina dispersoid had an average particle size of 0.09 micrometer and an interparticle spacing of 3.0 micrometer.

CONCLUDING REMARKS

Work at NASA has emphasized the use of a comminution and blending approach for the development of dispersion-strengthened materials. New processing methods are constantly being sought to produce improved products. A particular area of interest has been the production of fine clean powders which are required for production of high-strength dispersion-strengthened products.

If a triple-stirrer attritor with a bottom tap is used, it is now possible to produce fine composite powders in less time than with a single-stirrer attritor. This reduces the time available for segragation of the powder blend. This in turn reduces subsequent oxide agglomeration. It is possible not only to grind powders faster than with a single attritor, but also to separate the slurry from the grinding balls and media more quickly. The grinding alone takes about two-thirds the time for a single-stirrer attritor (69 as against 100 hr) to produce ultrafine powder blends of about equal quality. The greatest saving in time comes from the use of the bottom tap. Previously the entire charge of the mill had to be poured into containers and the powders separated from the balls and the grinding media. This would require 4 to 5 days. The bottom tap permits separation of the powders and cleaning of the container and grinding balls in about 4 to 8 hours.

Also of interest in the production of the $Ni\text{-}2Al_2O_3$ dispersion product is the use of a vacuum treatment subsequent to the hydrogen cleaning in the precleaning step. This approach presumably helps to remove the last traces of water vapor formed during the cleaning process. These and other processing modifications can be used to further improve the properties of dispersion-strengthened nickel as well as other matrix-oxide materials.

SUMMARY OF RESULTS

This investigation was conducted to determine whether nickel-alumina ($Ni\text{-}Al_2O_3$) dispersion-strengthened composites with an improved microstructure and with short-time tensile strengths comparing favorably with those of commercial thoriated nickel could be made from comminuted and blended powders. Improved processing methods including the use of a modified triple-stirrer attritor to grind powders and modified

powder precleaning methods were used for the study. The following results were obtained in the investigation.

1. A relation between the microstructural features and short-time tensile strength of the Ni-Al_2O_3 materials produced was observed. The best material produced had an average alumina particle size of 0.4 micrometer, an interparticle spacing of 0.7 micrometer, and a 1093° C (2000° F) tensile strength of 117 meganewtons per square meter (16 900 psi). This tensile strength exceeds that of the reference material, which was 96.5 meganewtons per square meter (14 000 psi), and is equivalent to that of commercially available thoriated sheet products.

2. A triple-stirrer attritor utilized for this program proved to be more effective in producing superior Ni-2Al_2O_3 composites than did a single-stirrer attritor used previously.

3. A precleaning treatment of the powders involving vacuum cleaning subsequent to hydrogen cleaning proved superior to treatments which involved hydrogen alone or vacuum preceding the hydrogen.

4. The thermomechanical processing method utilized in this study involved cold-working followed by intermediate anneals. The maximum in tensile strengths was obtained at 14 cycles for all conditions studied.

Lewis Research Center,
National Aeronautics and Space Administration,
Cleveland, Ohio, February 25, 1972,
134-03.

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TABLE I. - PRECLEANING SCHEDULES

[Temperature, 315° C (600° F).]

Material	Atmosphere	Time, hr
A[a]	Hydrogen	2
B	Hydrogen	2
C	Hydrogen and vacuum	1 1
D	Vacuum and hydrogen	1 1

[a]Material A schedule was used in previous study (ref. 5).

TABLE II. - 1093° C TENSILE TEST DATA FOR NICKEL SHEET DISPERSION-STRENGTHENED WITH 2 VOLUME PERCENT ALUMINA

[Precleaning treatment temperature, 315° C (600° F).]

Material	Attritor	Precleaning and annealing	Cold-roll - anneal cycles	Ultimate tensile strength at 1093° C	
				MN/m^2	psi
A[a]	Single stirrer	In hydrogen for 2 hr	7	55	8 000
			7	79	11 500
			14	86	12 500
			14	96.5	14 000
			18	76	11 000
			18	86	12 500
B	Triple stirrer	In hydrogen for 2 hr	7	99.5	14 400
			7	102	14 800
			14	103	15 000
			14	108	15 600
			21	76	11 000
			21	79	11 500
C	Triple stirrer	In hydrogen for 1 hr and vacuum for 1 hr	7	92	13 300
			14	117	16 900
			21	76	11 000
D	Triple stirrer	In vacuum for 1 hr and hydrogen for 1 hr	7	90	13 100
			14	106	15 400

[a]Data taken from ref. 5.

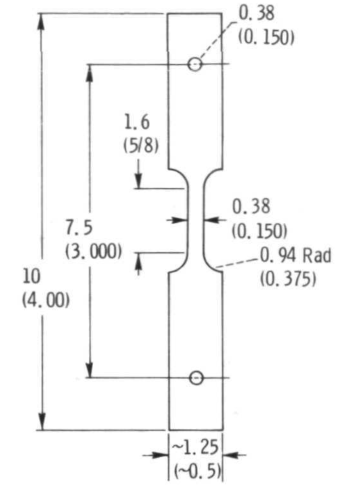


Figure 1. - Specimen configuration. (All dimensions are in centimeters (in.).)

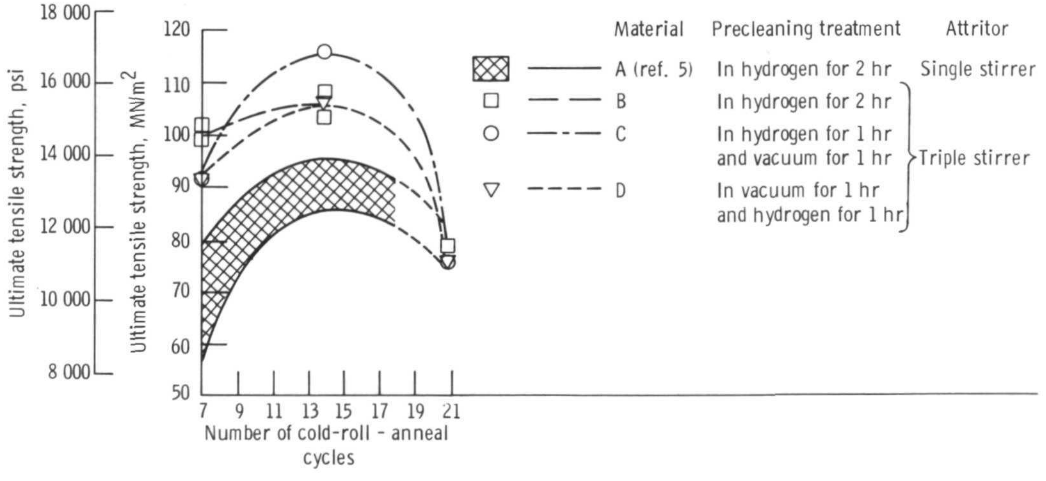
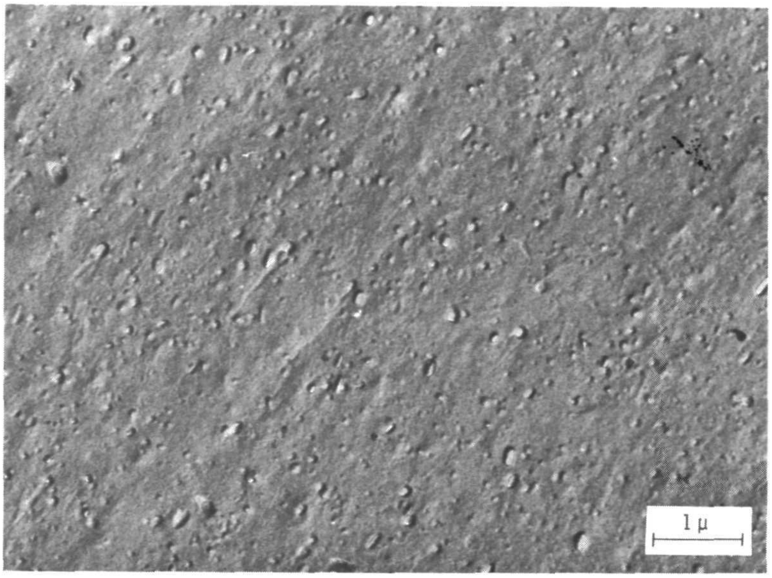
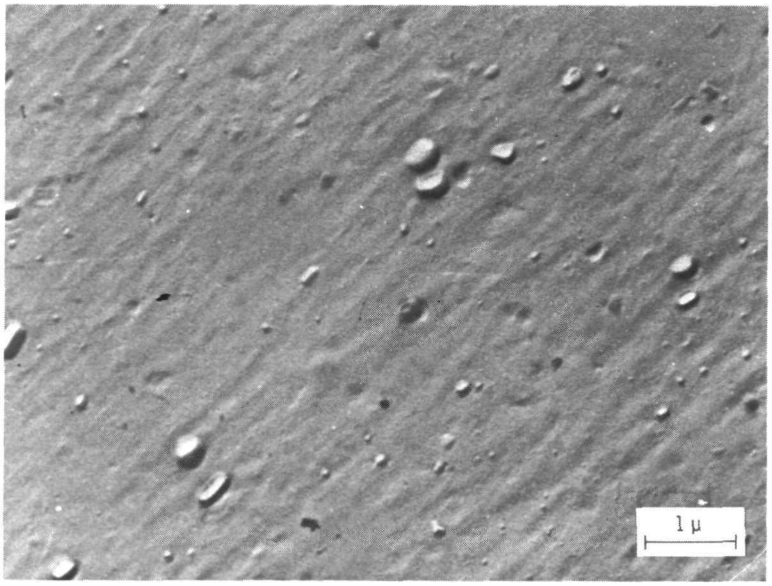


Figure 2. - Effect of processing variables on 1093° C (2000° F) tensile strength of Ni-2Al_2O_3 composite. Precleaning treatment temperature, 315° C (600° F).

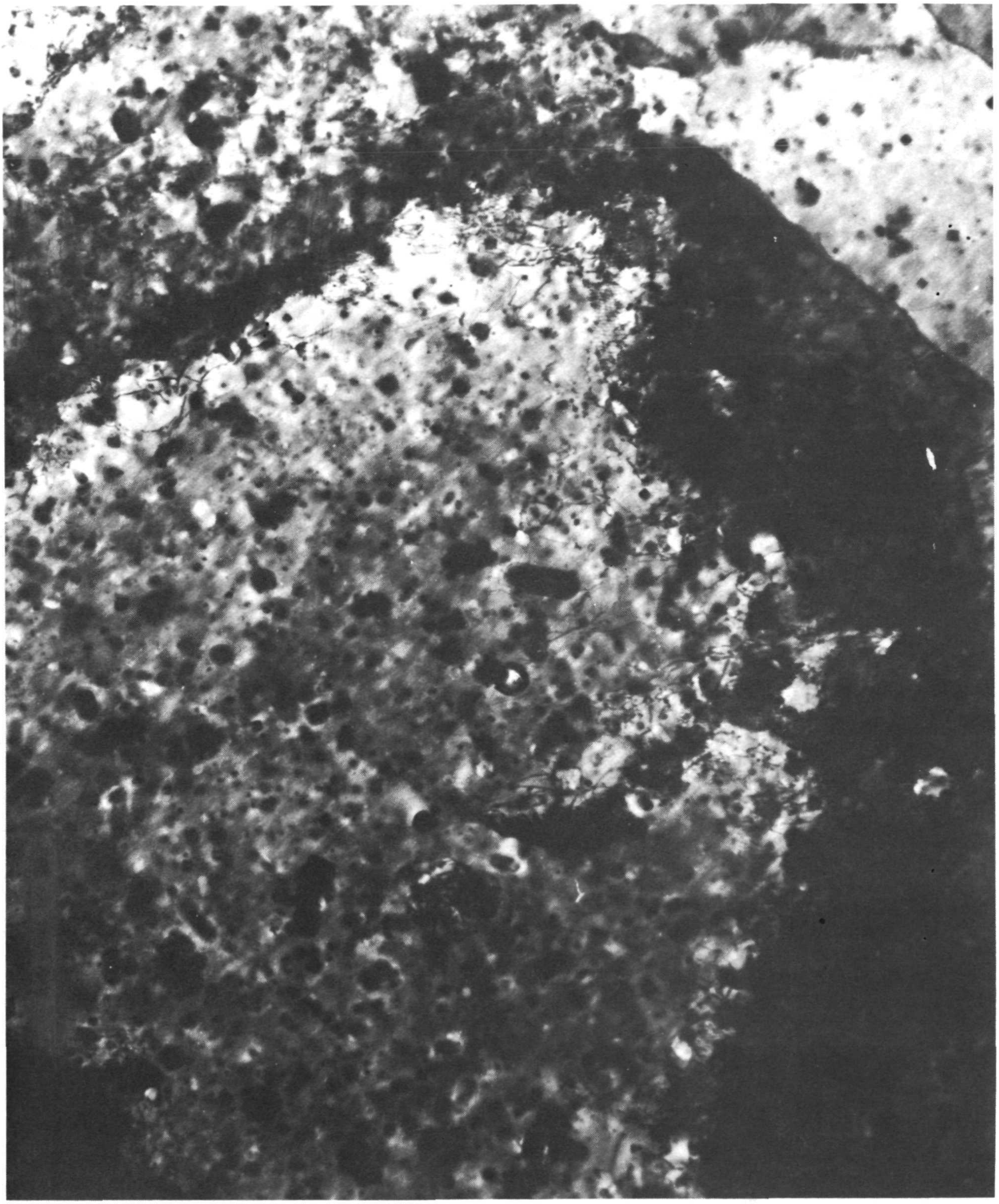


(a) Ultimate tensile strength, 117 meganewtons per square meter (16.9 ksi); ground in triple-stirrer attritor; precleaned in hydrogen for 1 hour and vacuum for 1 hour.



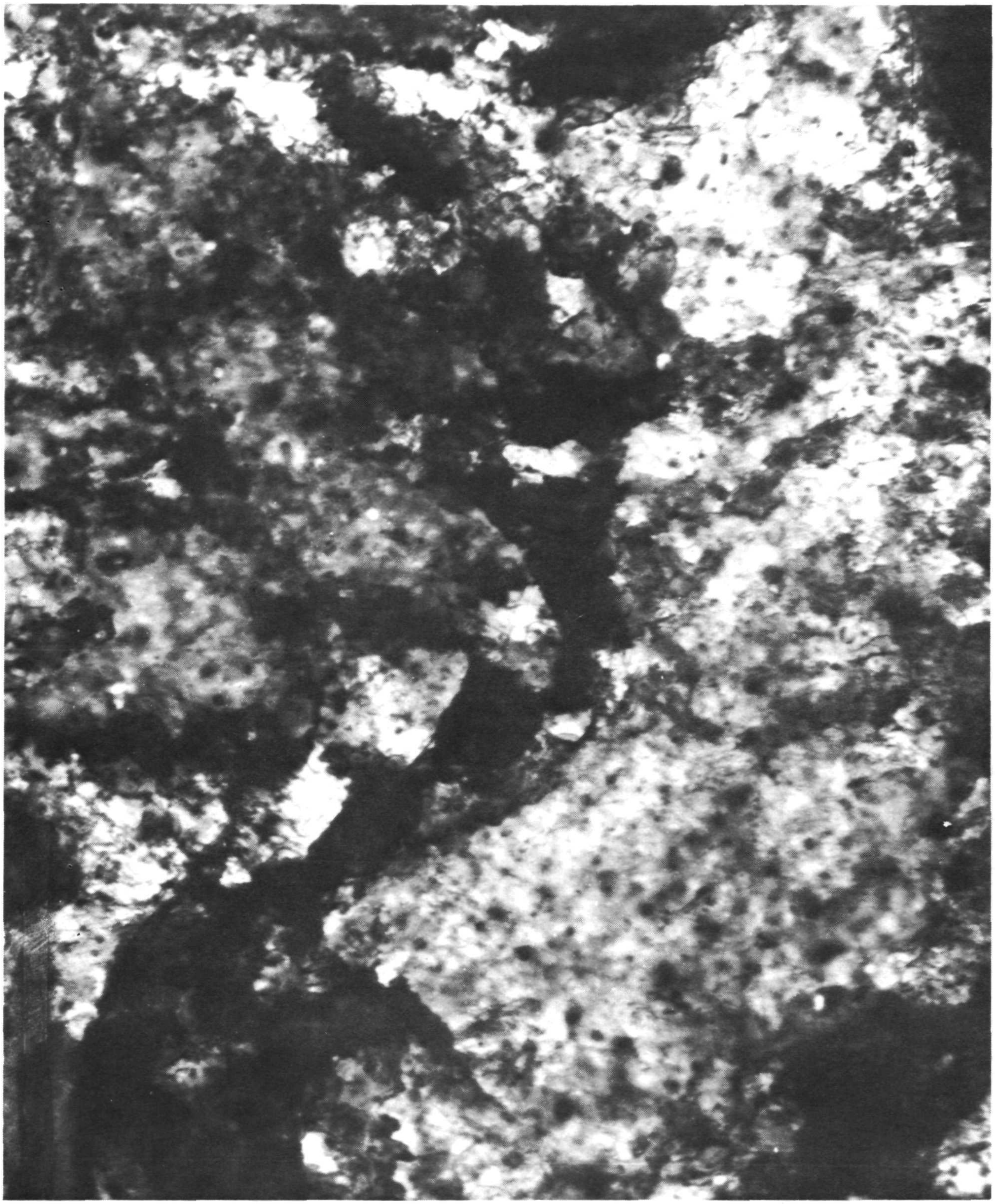
(b) Ultimate tensile strength, 79.1 meganewtons per square meter (11.3 ksi); ground in single-stirrer attritor; precleaned in hydrogen for 2 hours.

Figure 3. - Comparison of microstructures of Ni-2Al_2O_3 composites ground in single- and triple-stirrer attritors. Test temperature, 1093° C (2000° F); precleaning temperature, 315° C (600° F). X11 000.



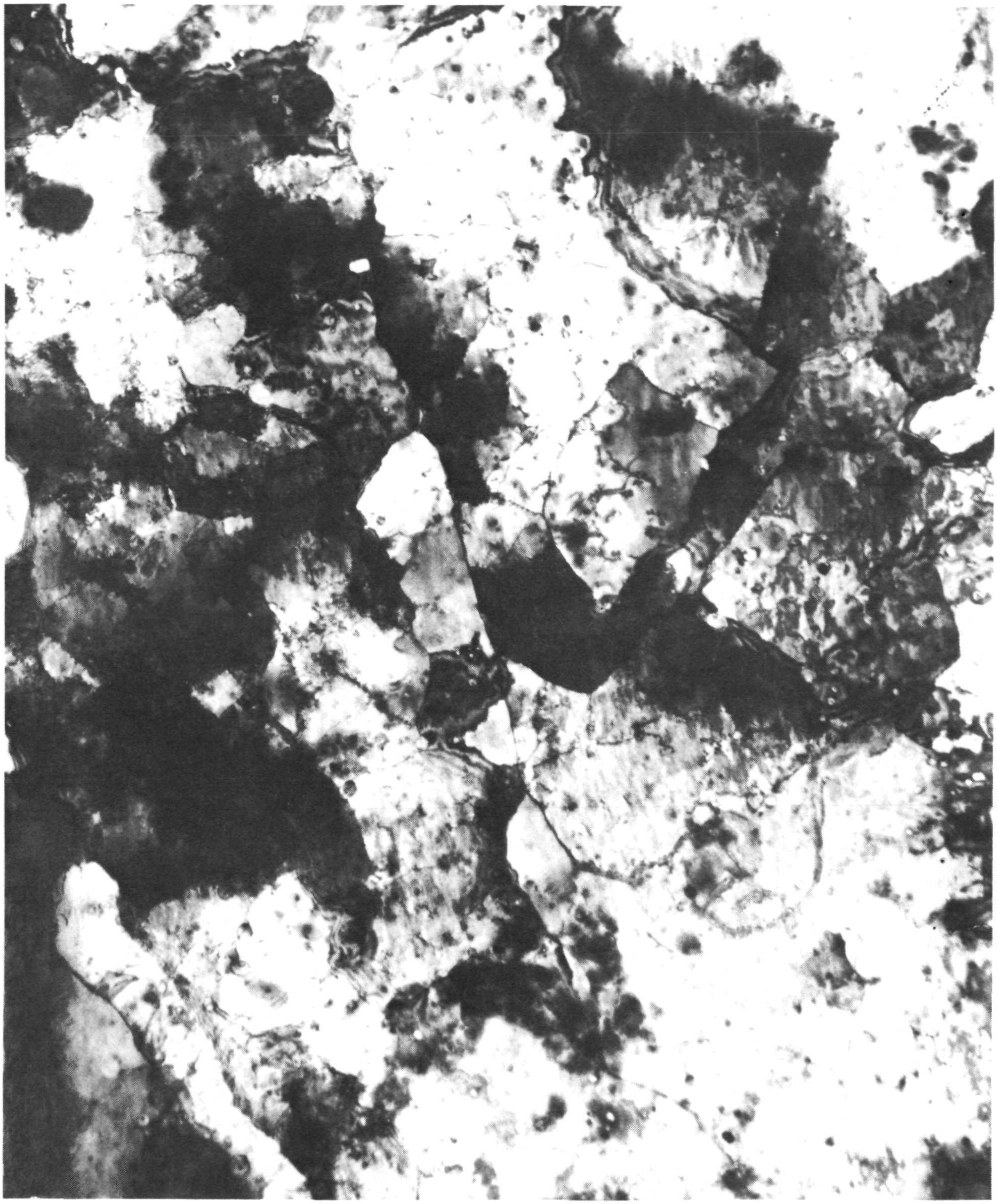
(a) Seven cold-roll - anneal cycles.

Figure 4. - Comparison of transmission electron micrographs for various stages of thermomechanical processing of Ni-2Al_2O_3 composite (material C). X27 000.



(b) Fourteen cold-roll - anneal cycles.

Figure 4. - Continued.



(c) Twenty-one cold-roll - anneal cycles.

Figure 4. - Concluded.

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